

Claims

1. A process for the preparation of a synthetic magnesium silicate having a crystal structure similar to natural hectorite, wherein the process comprises the steps of a) forming a precursor slurry, b) subjecting said precursor slurry to a continuous hydrothermal reaction in a pipe reactor at a temperature of from 210 to 400°C and under a pressure of at least 20 bar for 10 seconds to 4 hours, and c) washing and filtering to remove water soluble salts formed in the preparation of the precursor slurry, characterised in that said precursor slurry is not washed and filtered before it is subjected to said continuous hydrothermal reaction.
2. A process as claimed in claim 1 for the preparation of a synthetic magnesium silicate of the formula:

$$[\text{Si}_8 (\text{Mg}_a \text{Li}_b \text{H}_c) \text{O}_{20} (\text{OH})_{4-y} \text{F}_y]^{z-} \cdot z\text{M}^+$$
 wherein a is 4.95 to 5.7, b is from 0 to 1.05, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, and M is Na⁺ or Li⁺, the process consisting essentially of the following sequential steps:
 Preparing a precursor slurry by:
 forming an aqueous suspension of magnesium carbonate, and
 forming a silica precipitate in the aqueous suspension magnesium carbonate, the proportions of magnesium provided by the magnesium carbonate and of silica precipitated in the suspension corresponding to that of the formula of said magnesium silicate,
 subjecting the precursor slurry formed in step a) to a continuous hydrothermal treatment in a pipe reactor at a temperature of from 210 to 400°C and under a pressure of at least 20 bar for a period of from 10 seconds to 4 hours to form crystals of said synthetic magnesium silicate, and
 washing and filtering the product formed in step b) to separate water soluble salts from said synthetic magnesium silicate crystals.
3. A process as claimed in claim 1, wherein the process consists essentially of the following sequential steps:

- (a) forming an aqueous slurry from
a water-soluble magnesium salt,
ii) sodium silicate,
iii) sodium carbonate or sodium hydroxide and
5 iv) material delivering lithium and fluoride ions selected from the group consisting of
(A) lithium fluoride and (B) a lithium compound in conjunction with hydrofluoric acid,
fluosilicic acid, sodium silicofluoride all sodium fluoride, such that in the slurry the
following atomic ratios are present

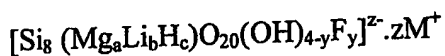
$$\begin{array}{rcl} \text{Si} & = & 0.5 \text{ to } 5.1 \\ \text{F} & & \text{Mg} \end{array} \qquad \begin{array}{rcl} \text{Li} & = & 0.1 \text{ to } 1.0 \\ & & \text{Mg} \end{array}$$

$$\begin{array}{rcl} \text{Si} & = & 0.5 \text{ to } 1.5 \\ \text{Mg} + \text{Li} & & \end{array} \qquad \begin{array}{rcl} \text{Na} & = & 1.0 \text{ to } 2.0 \\ 2 \text{ Mg} + \text{F-Li} & & \end{array}$$

- 15 the aqueous slurry being formed by co-precipitation by slowly combining the said
magnesium salt and the said sodium silicate and the said sodium carbonate or sodium
hydroxide, with heating and agitation, in an aqueous medium which contains the said
material or materials delivering the lithium and fluoride ions;
(b) taking the aqueous slurry so formed and, without washing free from soluble salts,
20 hydrothermally treating it in a pipe reactor at a temperature of from 210 to 400°C and
under a pressure of at least 20 bar for 10 seconds to 4 hours to form synthetic
magnesium silicate crystals, and
(c) washing and filtering the product formed in step b) to separate water soluble salts
from said synthetic magnesium silicate crystals.

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4. A process as claimed in claim 1 for the preparation of a synthetic magnesium silicate
of the formula:



- wherein a is 4.95 to 5.7, b is from 0 to 1.05, c is from 0 to <2, a+b+c is from 5 to <8, y
30 is from 0 to <4, z = 12-2a-b-c, and M is Na⁺ or Li⁺, the process consisting essentially of
the following sequential steps:

- precipitating a magnesium silicate having the desired value of "a" in a slurry by combining an aqueous solution of a water soluble magnesium salt with an aqueous alkaline solution of one or more sodium compounds in the presence of dissolved silicon-delivering material, the pH of the alkaline solution being maintained at 8 to 12.5 throughout,
- 5 without first drying or washing, hydrothermally treating the aqueous slurry formed in a) in a pipe reactor at a temperature of from 210 to 400°C and under a pressure of at least 20 bar for 10 seconds to 4 hours to form synthetic magnesium silicate crystals, and washing and filtering the product formed in step b) to separate water soluble salts from
- 10 said synthetic magnesium silicate crystals.
5. A process as claimed claim 1, wherein the hydrothermal treatment step b) is conducted in a pipe reactor at a temperature of from 240 to 380 °C and at a pressure of at least 30 bar, more preferably at a temperature of from 250 to 350°C and at a pressure
- 15 of at least 40 bar.
6. A process as claimed in claim 5, wherein the temperature is in the range of from 285 to 315°C and the pressure is at least 70 bar.
- 20 7. A process as claimed in claim 1, followed by drying the the synthetic magnesium silicate crystals under normal atmospheric pressure at a temperature up to 450°C after they have been washed and filtered in step c).
8. A process for the preparation of a precursor slurry intended for use in the process
- 25 claimed in claim 1, wherein the process for preparing said slurry is a continuous process conducted at a temperature of up to 400°C.
9. A process as claimed in claim 8, wherein a pipe reactor is employed for the continuous preparation of the precursor slurry.
- 30 10. A process as claimed in claim 1, wherein the precursor slurry is formed by a continuous process conducted at a temperature of up to 400°C.

11. A process as claimed in claim 10, wherein said continuous process is conducted in a pipe reactor.

5 12. A process as claimed in claim 11, wherein both the formation of the precursor slurry and the hydrothermal reaction take place simultaneously as a continuous process in a single pipe reactor.

13. A process as claimed claim 2, wherein the hydrothermal treatment step b) is
10 conducted in a pipe reactor at a temperature of from 240 to 380 °C and at a pressure of at least 30 bar, more preferably at a temperature of from 250 to 350°C and at a pressure of at least 40 bar.

14. A process as claimed in claim 13, wherein the temperature is in the range of from
15 285 to 315°C and the pressure is at least 70 bar.

15. A process as claimed in claim 2, followed by drying the the synthetic magnesium silicate crystals under normal atmospheric pressure at a temperature up to 450°C after they have been washed and filtered in step c).

20 16. A process for the preparation of a precursor slurry intended for use in the process claimed in claim 2, wherein the process for preparing said slurry is a continuous process conducted at a temperature of up to 400°C.

25 17. A process as claimed in claim 16, wherein a pipe reactor is employed for the continuous preparation of the precursor slurry.

18. A process as claimed in claim 2, wherein the precursor slurry is formed by a continuous process conducted at a temperature of up to 400°C.

30 19. A process as claimed in claim 18, wherein said continuous process is conducted in a pipe reactor.

20. A process as claimed in claim 19, wherein both the formation of the precursor slurry and the hydrothermal reaction take place simultaneously as a continuous process in a single pipe reactor.

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21. A process as claimed claim 3, wherein the hydrothermal treatment step b) is conducted in a pipe reactor at a temperature of from 240 to 380 °C and at a pressure of at least 30 bar, more preferably at a temperature of from 250 to 350°C and at a pressure of at least 40 bar.

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22. A process as claimed in claim 21, wherein the temperature is in the range of from 285 to 315°C and the pressure is at least 70 bar.

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23. A process as claimed in claim 3, followed by drying the the synthetic magnesium silicate crystals under normal atmospheric pressure at a temperature up to 450°C after they have been washed and filtered in step c).

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24. A process for the preparation of a precursor slurry intended for use in the process claimed in claim 3, wherein the process for preparing said slurry is a continuous process conducted at a temperature of up to 400°C.

25. A process as claimed in claim 24, wherein a pipe reactor is employed for the continuous preparation of the precursor slurry.

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26. A process as claimed in claim 3, wherein the precursor slurry is formed by a continuous process conducted at a temperature of up to 400°C.

27. A process as claimed in claim 26, wherein said continuous process is conducted in a pipe reactor.

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28. A process as claimed in claim 27, wherein both the formation of the precursor slurry and the hydrothermal reaction take place simultaneously as a continuous process in a single pipe reactor.
- 5 29. A process as claimed claim 4, wherein the hydrothermal treatment step b) is conducted in a pipe reactor at a temperature of from 240 to 380 °C and at a pressure of at least 30 bar, more preferably at a temperature of from 250 to 350°C and at a pressure of at least 40 bar.
- 10 30. A process as claimed in claim 29, wherein the temperature is in the range of from 285 to 315°C and the pressure is at least 70 bar.
31. A process as claimed in claim 4, followed by drying the the synthetic magnesium silicate crystals under normal atmospheric pressure at a temperature up to 450°C after
15 they have been washed and filtered in step c).
32. A process for the preparation of a precursor slurry intended for use in the process claimed in claim 4, wherein the process for preparing said slurry is a continuous process conducted at a temperature of up to 400°C.
- 20 33. A process as claimed in claim 32, wherein a pipe reactor is employed for the continuous preparation of the precursor slurry.
34. A process as claimed in claim 4, wherein the precursor slurry is formed by a
25 continuous process conducted at a temperature of up to 400°C.
35. A process as claimed in claim 34, wherein said continuous process is conducted in a pipe reactor.
- 30 36. A process as claimed in claim 35, wherein both the formation of the precursor slurry and the hydrothermal reaction take place simultaneously as a continuous process in a single pipe reactor.